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ELECTRON MICROSCOPE STUDIES ON THE COTTON CELLULOSE

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ABSTRACT Hydrolysis of cotton cellulose in strong mineral acid shows a disintegrating fibrillar structure under electron microscope. The broken up fibrils and particles have almost the same lateral dimension and there is an indication of layer lattice structure of cellulose. No definite minimum of the length of the particles could be observed.

INTRODUCTION

Morphological study of cotton cellulose by some workers (Balls and Hancock, 1922) under light microscope shows dislocated striations. Dislocation of striations were also noted in most of the bast fibres prominently in flax (Muhlethaler, 1949). The transverse section shows cell structure in both the varieties (Hock *et al.*, 1940). The molecular structure has been studied by X-ray and the unit cell has been defined (Hessler *et al.*, 1948). The state of orientation and purity of fibre, so far as α-cellulose content in fibre is concerned, has also been studied (Berkley *et al.*, 1938, 1949).

From the usual X-ray diagram and also from small angle scattering, the average length of cellulose crystallites of Remie was found to vary between 500 Å and 600 Å (Heyn, 1950). But the exact shape and locations of these crystallites in the fibre could not be ascertained for want of proper technique.

It will be observed that the size of crystallites as suggested from X-ray studies is within the range which could be advantageously studied under electron microscope. The early attempts to study the structure of cellulose with electron microscope by Ruska and others (1940) did not succeed. Subsequently, attempts by replica technique by Astbury and Preston (1948) indicated the fibrillar structure, while Freywissling (1948) using mechanical grinding reported fibrillar structure of varying width and length. Subsequently Ranbi and Ribi (1949 and 1951) and also Mukherjee and Woods (1953) tried acid hydrolysis for the disintegration of fibres. They were successful in breaking the fibrils into descrete particles, believed to be the crystallites of cellulose. The present work contains an account of the electron microscopic studies on Indian cotton cellulose using the acid hydrolysis technique developed by Mukherjee and Woods (1953).

EXPERIMENTAL PROCEDURE

In this work a sample of raw Indian cotton was dewaxed by soxhlate extraction with Carbon tetra-chloride and after drying, it was further purified by boiling in dilute (2%) sodium hydroxide solution for 4 hours. The purified fibre was next treated in sulphuric acid solution of strength 920 grammes per litre at 32°C. The fibres disintegrated into small fragments and dispersed in the acid. In an attempt to wash this material by distilled water in a centrifuge, the fibres dispersed still further in a colloidal solution at a pH round about 4. The colloidal solution obtained by peptisation at each washing in the centrifuge was collected. This acidic colloidal solution was then dialysed in cellophen bags in distilled water at room temperature. Four to five days had to be allowed in the process of dialysis to raise the *p*H of the solution to about 5.5. Because the *p*H of distilled water itself against which the solution was dialysed was 5.8. The dialysed solution was further diluted with distilled water in order to obtain a concentration suitable for electron microscope. The specimen for an electron microscope was obtained by putting a small drop of solution on a colloidion coated microscope grid and evaporating the water to leave the cellulose behind. The specimen was next shadowed with chromium and subsequently examined under electron microscope. The microscope used was Siemens' Elmiskop I at 60kV.

TABLE I

Obs. nos.	Width of individual particles on the micrograph (cm)	Actual width of individual particles (cm) ÷ 17000
1	1.96×10^{-2}	115.3×10^{-8}
2	2.91	171.2
3	1.97	115.9
4	2.89	170.0
5	1.79	105.3
6	2.13	125.3
7	2.06	121.2
8	1.82	107.1
9	2.83	166.5
10	1.81	106.5
Mean width = 130.4±8.70 Å.		

DISCUSSION

The electron micrograph (Fig. 1) shows a distinct fibrillar structure with a definite indication of breakdown into elongated discrete particles as could be

seen in the field of observation. It is also observed on the micrograph that the fibrils and the particles have more or less the same lateral dimension. In other

Fig 1.

words the structure as revealed under microscope shows a disintegrating fibrillar structure breaking into particles. On closer observation of the aggregates, there is an indication that they are rather in flat layers which probably are in conformity with the idea of layer lattice, associated with structure of cellulose. No definite minimum of the length of the particle could be observed. But they were found to vary from 500 Å to 2500 Å and above. Nevertheless the width of the particles was found to vary within a narrow range, when measured. The average width of the particle was found to be about 130.4 ± 8.70 Å.

It is expected that under favourable conditions, by using different time, concentration and temperature of acid it may be possible to study the individual units (fibrils) which is the basis of building mechanism of the structure. This work is being further pursued to see if similar results can be obtained in case of bast fibres and to study the difference, if any, between the bast fibres and comparatively more pure varieties of fibres like cotton and also amongst the bast fibres themselves.

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